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Publication number : **0 636 689 A2**

(12)

EUROPEAN PATENT APPLICATION

(21) Application number : **94305450.2**

(22) Date of filing : **22.07.94**

(51) Int. Cl.⁶ : **C11D 3/39, C11D 3/395,
C11D 3/37, C11D 1/14**

(30) Priority : **30.07.93 GB 9315761**

(43) Date of publication of application :
01.02.95 Bulletin 95/05

(84) Designated Contracting States :
**AT BE CH DE DK ES FR GB GR IE IT LI LU MC
NL PT SE**

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(84) **BE CH DE DK ES FR GR IT LI LU MC NL PT SE
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(54) **Bleach compositions.**

(57) A bleach composition comprises an active halogen or a peroxy bleach material, preferably hypochlorite; surfactant constituted by at least 50% w/w primary alkyl sulphate; and polymeric thickener comprising charged, hydrophilic monomer and uncharged hydrophobic monomer. The polymeric thickener preferably comprises a copolymer of methacrylic acid and styrene having proportions (wt%) of methacrylic acid and styrene ranging from 80:20 to 20:80, and preferably comprises 54 wt% methacrylic acid and 46% styrene, with a molecular weight of about 100,00 and substantially no cross-linking. The polymeric thickener acts to enhance thickening and enables use of a wider range of surfactants than hitherto possible.

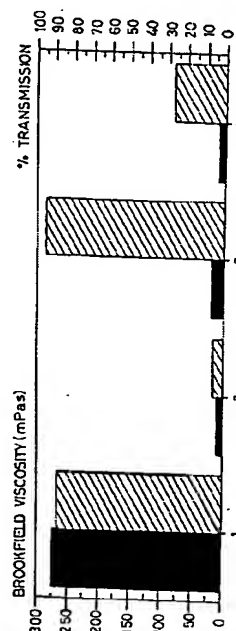


Fig. 1

EP 0 636 689 A2

Field of the Invention

This invention concerns bleach compositions, particularly thickened aqueous bleach compositions.

5 Background to the Invention

There is much prior art concerning thickened aqueous bleach compositions, using a variety of thickening agents. See, for example, EP 0256638 (Clorox), EP 0373864 (Procter & Gamble) and EP 0329419 (Unilever) and the prior art referred to in those documents.

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Summary of the Invention

According to the present invention there is provided a bleach composition, comprising an active halogen or a peroxy bleach material; surfactant; and polymeric thickener comprising charged, hydrophilic monomer and uncharged, hydrophobic monomer, said surfactant comprising at least 50% w/w primary alkyl sulphate.

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The charged, hydrophilic monomer is preferably selected from group: acrylic acid and substituted acrylic acids, eg. methacrylic acid, maleic acid and half-esters thereof, crotonic and itaconic acids and other alkene carboxylic acids and their derivatives, carboxylated styrene, sulphonated styrene, ethylene sulphonic acid, and certain nitrogen-based monomers eg acrylamide, vinyl pyrrolidone, and mixtures of such hydrophilic monomers.

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The uncharged hydrophobic monomer is preferably selected from the group: styrene and substituted styrenes eg. alpha-methyl styrene, alpha-olefins, eg. C₁₂-alpha-olefin, vinyl ethers, eg. butyl vinyl ether; allyl ethers, eg. butyl allyl ether; unsaturated hydrocarbons, eg ethylene, propylene, cyclohexene and dienes, eg. 1,6 hexadiene, divinyl benzene, cyclohexadiene. Such hydrophobic moieties may also be introduced into the polymer by chain transfer agents and initiators, eg. mercaptans, eg. dodecyl mercaptans, and peroxides and azo initiators, eg. dilauryl peroxide, AZBN (azobisisobutyronitrile). Mixtures hydrophobic monomers may also be used.

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A single monomer with both hydrophilic and hydrophobic properties may also be used.

The preferred polymer comprises a copolymer of methacrylic acid and styrene, conveniently having proportions (wt %) of methacrylic acid and styrene ranging from 80:20 to 20:80.

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Additional monomers, such as acrylic esters, acrylates, olefins and substituted styrenes, may optionally be included, eg in place of up to about half of the amount of styrene.

The polymer is preferably substantially non cross-linked, for reasons that will be explained below, although a degree of cross-linking can be tolerated.

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The polymer preferably has a molecular weight of about 100,000, although this is not critical and indeed it can be difficult to produce a polymer having an accurately specified molecular weight. Molecular weights in the range 80,000 to 120,000 are thus regarded as about 100,000. If the molecular weight is substantially lower, then this has a deleterious effect on thickening properties of the polymer. Further, if the molecular weight of the polymer is too high, then clarity and stability of the composition can be adversely affected.

40

One particularly preferred polymer comprises 54 wt% methacrylic acid and 46% styrene, with a molecular weight of about 100,000 and substantially no cross-linking. Such a polymer is found to be hypochlorite-stable and to be an effective thickener in hypochlorite bleach.

Such polymers can be readily made in conventional manner, eg by emulsion polymerisation.

The polymer is present in an appropriate amount to achieve thickening to a desired degree, and is typically present in an amount from 0.1 to 7.5% by wt as active material, preferably 0.5 to 3% by wt as active material. Suitable amounts for any particular composition can be readily determined by experiment, depending on bleach concentration, target viscosity and other considerations. In general, the greater the concentration of bleach material, the greater the amount of polymer (and also surfactant) required to achieve a given viscosity.

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The bleach material may comprise one or more peroxy compounds, as are well known in the art, including peracids and salts thereof, and hydrogen peroxide and derivatives thereof. Preferably, however, the bleach material comprises one or more halogen compounds, as are also well known in the art, including alkali metal and alkaline earth salts of hypochlorite, haloamines, haloimines, haloimides and haloamides. Hypochlorite and compounds producing hypochlorite in aqueous solution are preferred. Particularly preferred is sodium hypochlorite, NaOCl, typically in an amount ranging from 0.2 to 15% by weight, more preferably 0.2 to 10% by weight, and typically providing an amount of available chlorine in the range 1 to 5% by wt.

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The surfactant primary alkyl sulphate (PAS), which is sometimes also referred to as primary alcohol sulphate or primary alkyl sulphate, is an environmentally acceptable surfactant, hence use of this material to constitute at least 50% by weight of the surfactant of the composition of the invention.

The surfactant has a cleaning function, and it is possible to use one or more surfactants with PAS, selected from a wide range, including the following: alkyl ether sulphates, eg. lauryl ether sulphate such as those sold under the Trade Names Empicol ESB3, Empicol MD, and Perlankrol ESD, alcohol sulphates, tall w alcohol sulphate such as those sold under the Trade Name Empicol TAS30, alkyl benzen sulphonates, eg that sold under the Trade Name Nansa SL30, paraffin sulphonates, eg that sold under the Trade Name Lutensit A-PS, alkyl phenol ether sulphates, eg. those sold under the Trade Names Perlankrol PA conc, Perlankrol SN, and Perlankrol RN75, alcohol ethoxylates, eg. those sold under the Trade Names Dobanol 91-6, Ethylan CD 91-6, Empilan KC8 and Synperonic A7, nonylphenol ethoxylates, eg that sold under the Trade Name Ethylan BCP, sodium carboxylates (soaps) (salts of fatty acids), manufactured from fatty acids such as those sold under the Trade Names Prifac 7908, and Prifac 6920, by neutralisation of the acid with a base eg sodium hydroxide or triethanolamine; alkyl ether carboxylates, eg that sold under the Trade name eg. Olin CS1, alkyl polyglucosides, eg. those sold under the Trade Name Plantaren, EP/PO block copolymers, eg those sold under the Trade Plurafac, Triton, and Monolan PC, alkyl diphenyloxide sulphonate eg. that sold under the Trade Name Dowfax 3B2, fatty acid amides, eg those sold under the Trade Names Empilan CME/CDE, acyl sarcosinates, alkyl taurides, sulphosuccinates, eg that sold under the Trade Name Empimin OT, alpha-olefin sulphonates, phosphate esters (alkyl and/or aryl), eg those sold under the Trade Name Phospholan, ethoxylated fatty acids.

Suitable mixtures of surfactants are generally found to give good results.

The composition is preferably substantially free of nonionic surfactants (which tend to have a high adverse environmental impact, especially in the case of ethoxylated surfactants) and nitrogen-based surfactants (which can also be environmentally detrimental, especially in the case of amine oxides, which are highly reactive).

Good results have been obtained with lauryl ether sulphates, eg those sold under the Trade Names Empicol ESB3 (lauryl ether sulphate, Na salt, with 2 ethylene oxide units (2EO)), and Empicol MD (lauryl ether sulphate, Na salt, with 4 EO). A mixture of these two surfactants, eg in wt proportions of active matter of MD: ESB3 of 2:1, is found to give particularly good results in a hypochlorite bleach composition.

Particularly good results have been obtained with mixtures of primary alkyl sulphates (PAS) and linear ether sulphates (LES), conveniently in wt proportions of active matter of PAS:LES of 2:1. Use of such a mixture with relatively low levels (about 2% by wt active material) of polymer provides a composition with good viscosity, clarity and cleaning properties.

Surfactant is conveniently present in an amount in the range 0.5 to 5% by wt active matter, typically about 2% to 3% by wt active matter.

The composition may optionally include electrolyte. Low levels of electrolytes such as sodium chloride function to provide ions in aqueous solution and have been shown to improve solution viscosity. Sodium chloride is generally present in sodium hypochlorite as available commercially, or may be added to the composition in appropriate amounts.

The composition may also optionally include buffer, to maintain pH. Alkaline pHs, typically between 11 and 14, eg about 13, are generally appropriate to achieve desired viscosity and stability. Some reagents function both as electrolyte and buffer.

Further optional ingredients such as fragrance, colouring agents, whiteners, solvents and builders may be included for aesthetic reasons or to improve properties.

One preferred composition in accordance with the invention comprises hypochlorite bleach, the preferred methacrylic acid/styrene polymer discussed above (having 54% methacrylic acid and 46% styrene, a molecular weight of about 100,000 and being substantially non-cross linked), and a mixture of lauryl ether sulphate surfactants, particularly Empicol ESB3 and Empicol MD. This composition is a clear, viscous, stable, isotropic formulation, with good cleaning and bleaching properties and good stability. It is thought the polymer is acting as a hydrotrope, and thickening arises due to the interaction of the polymer with the surfactant micelles and not solely from the swelling of the polymer.

As mentioned above, it is preferred to use substantially non cross-linked polymer. Non cross-linked polymers are found to give compositions with better clarity than those using cross-linked polymer, particularly if perfume (which is generally an oil) is present in the composition. This observation has not been fully explored, but it is thought that there is a competitive interaction between polymer & surfactant and surfactant & perfume.

By using substantially non cross-linked polymer it is also found that the polymer assists the solubilisation of perfume in the composition when cold, and that it is not necessary to warm the composition for the perfume to dissolve: the ability to process in the cold is a significant manufacturing advantage.

Furthermore, a synergistic effect has been observed between surfactant and polymer that is also thought to be related to the state of cross-linking of the polymer: hypochlorite bleach containing both polymer and surfactant is found to have substantially improved characteristics of viscosity and clarity as compared with hypochlorite containing only polymer or only surfactant. This effect is not fully understood, but is much more marked

with substantially non cross-linked polymer, and it is thought that the polymer reacts with the surfactant in some way, as discussed above.

Compositions in accordance with the invention, in preferred embodiments at least, have certain advantages over similar prior art compositions, including the following:

- 5 1. They are more substantially free of nitrosamines commonly associated with compositions using nitrogen-based surfactants, such as amine oxides.
2. A wider range of co-surfactants can be used than has hitherto been possible, enabling surfactants to be selected for their cleaning properties. By way of comparison, amine oxide can only be used with a restricted range of surfactants to achieve desired viscosities.
- 10 3. The polymer acts to solubilise perfumes, as discussed above.

The compositions of the invention find particular, but not exclusive, application in hard surface cleaning, e.g. of bathroom and kitchen surfaces, and may be embodied as hard surface cleaners, multisurface cleaners, toilet cleaners or drain cleaners.

The invention will be further described, by way of illustration, in the following examples and by reference to the accompanying drawings, in which:

Figure 1 is a series of graphs of viscosity and % transmission for various mixtures, with results for viscosity shown by solid bars and those for % transmission shown by hatched bars; and

Figure 2 is a further series of graphs similar to Figure 1.

20 Example 1 Manufacture of Methacrylic Acid/Styrene Polymer

A substantially non-cross linked polymer (referred to as polymer A) of methacrylic acid (54% wt) and styrene (46% wt) with a molecular weight of about 100,000 (characterised by GPC compared with polyacrylate standards) was made on a pilot scale by conventional emulsion polymerisation techniques. The materials used were as follows:

	<u>Grms</u>	<u>Remarks</u>
30 <u>Reactor charge</u>		
Deionized water	4500	Heat to 80°C under nitrogen
Crodafos N3A	112.5	
35 .910 Ammonia soln.	37.5	
Empilan KB3	180	
40 <u>Initial monomer</u>		
Methacrylic acid	210	Add at 70°C hold a further 15 mins. once the reactor temperature reaches 80°C.
45 Styrene	150	
50		
55		

Initial catalyst

5	Potassium persulphate	3	Add at 70°C with
	Deionized water	280	init. mons.

Monomer (slow-add)

10	Deionized water	3750	Add over 3 hours at
	Crodafos N3A	112.5	80°C.
	Empilan KB3	180	
	Methacrylic acid	2250	
15	Styrene	1935	

Catalyst (slow-add)

20	Deionized water	1200
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Scavenge

25	Potassium persulphate	3	Add over 1 hour
	Deionized water	900	

30 The procedure used was as follows.

The initial charge of deionised water and surfactants were added to a 15 litre stainless steel reactor fitted with a lid which has inlet ports for an agitator, water condenser and for the addition of monomer and initiator solutions. The contents of the reactor were heated to 80°C under nitrogen.

35 At 70°C a portion of the monomer pre-emulsion and initiator solution were added to the reactor and were allowed to initiate. After initiation had taken place the remainder of the monomer pre-emulsion and the initiator solutions were added to the reactor concurrently over 3 hours and 3.5 hours respectively at 80°C. At the end of the initiator addition the reactor temperature was maintained at 80°C for a further 1 hour to eliminate any unreacted monomer. After completion of the 1 hour hold the emulsion polymer was cooled to less than 30°C, filtered and transferred to storage.

40 The resulting product was an aqueous dispersion of methacrylic acid/styrene copolymer, with the following typical properties.

45	Solids	= 31.75%
	pH	= 2.6
	Brookfield Viscosity (2/20rpm)	= 30mPas

Example 2. Behaviour of polymer A in 5% Sodium Hypochlorite

50 In order to test the behaviour of polymer A in 5% sodium hypochlorite, various mixtures were made up as follows (all % are w/w active matter):

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1.	Empicol MD	2%
	Empicol ESB3	1%
	Polymer A	1.74%
	NaOCl	5%
2.	Empicol MD	2%
	Empicol ESB3	1%
	NaOCl	5%

This mixture was non-homogeneous and separated overnight.

3.	Polymer A	1.74%
	Deionised water	balance to 100%
4.	Polymer A	1.74%
	NaOCl	5%

In all cases the pH was adjusted to 13 with NaOH.

For each mixture the % transmission at 500nm was tested to give an indication of clarity, and the viscosity at 3/20 rpm was also tested. The results are shown graphically in Figure 1.

In Figure 1, bars 1 indicate results for mixture 1 above, bars 2 results for mixture 2, bars 3 results for mixture 3 and bars 4 results for mixture 4.

It can be seen from Figure 1 that polymer or surfactant alone in hypochlorite (mixtures 4 and 2, respectively) give poor results in terms of both clarity and viscosity. However, mixtures of polymer, surfactant and hypochlorite (mixture 1) give very good results in terms of both clarity and viscosity. A synergistic effect is thus occurring between the polymer and surfactant.

Example 3. Effects of cross-linked polymers on viscosity and clarity in unperfumed hypochlorite

In order to test the effects of cross-linked polymers on viscosity and clarity in unperfumed hypochlorite, a crosslinked version of polymer A (known as polymer B) was prepared in conventional manner.

Various mixtures of this polymer B (1.74% by wt active), surfactant (3% by wt active Empicol ESB3) and sodium hypochlorite (1.6% by wt active) were made up, with pH adjusted to 13 with NaOH. The viscosity at 20 rpm and % transmission at 450 nm were tested, and the results are shown graphically in Figure 2.

The mixtures tested (with results in correspondingly numbered bars in Figure 2) were as follows:

1. Surfactant + polymer A in bleach
2. Polymer A in bleach
3. Surfactant + polymer A in water
4. Polymer A in water
5. Surfactant in bleach
6. Surfactant + polymer B in bleach
7. Polymer B in bleach
8. Surfactant + polymer B in water
9. Polymer B in water.

The results of Examples 2 and 3 show that cross-linked polymers generally give higher viscosities but lower clarities in hypochlorite than do non-cross-linked polymers. Further work (details not included) has shown that this effect is magnified in the presence of perfume/fragrance.

Similar experiments using 5% hypochlorite solutions show that cross-linked polymers are less compatible with these systems leading to cloudier solutions which are more unstable.

Based on this and other experimental data, a thickened bleach composition suitable for general domestic

use was prepared.

Example 4. Thickened bleach composition using polymer A

A thickened 5% hypochlorite formulation was made by mixing the following ingredients:

Component	Use Level (%)
	(% w/w active matter)
Empicol MD (30% active)	2
Empicol ESB3 (27.5% active)	1
Polymer A (31% active)	1.74
Sodium hypochlorite (14% active)	5
Fragrance	0.3%
Water	100%

pH was adjusted to 13 with 4% caustic.

The resulting composition had a clear aspect and a viscosity (Brookfield) 3/20 rpm of 275 mPas. and is suitable for use eg as a domestic multi-surface cleaning product.

After further tests with surfactant systems, a further optimised composition was produced.

Example 5. Thickened bleach composition using polymer A

A thickened sodium hypochlorite composition (having 1.3% available chlorine) was made by mixing the following ingredients:

Component	Use level (%)
	(% w/w active matter)
Polymer A (at 36%)	2.0%
Caustic (100 Twaddle i.e. 47%)	1.49%
Dacton L27 (Primary alkyl sulphate) (at 27%)	2.0%
Dobanol 23-35-27 (Linear ether sulphate) (at 27%)	1.0%
Dye premix	1.0%
Perfume	0.3%
Hypochloride (at 15.4% available Cl)	8.44%
	(1.3% AvCl)
NaCl (hypo. soln. is 12.8%)	10.8%
Alkaline silicate	0.107%
Water	to 100%

pH was adjusted to 13 with 40% caustic.

The resulting composition had a clear aspect, good viscosity, stability and cleaning properties, and is suitable for use, eg as a domestic multi-surface cleaning product.

Claims

1. A bleach composition, comprising an active halogen peroxide bleach material; surfactant; and polymeric thickener comprising charged, hydrophilic monomer and uncharged, hydrophobic monomer, said surfactant comprising at least 50% w/w primary alkyl sulphate.
2. A composition according to claim 1, wherein the charged, hydrophilic monomer is selected from group: acrylic acid and substituted acrylic acids, eg. methacrylic acid, maleic acid and half-esters thereof, crotonic and itaconic acids and other alkene carboxylic acids and their derivatives, carboxylated styrene, sulphonated styrene, ethylene sulphonic acid, and certain nitrogen-based monomers eg acrylamide, vinyl pyrrolidone, and mixtures of such hydrophilic monomers.
3. A composition according to claim 1 or 2, wherein the uncharged hydrophobic monomer is selected from the group: styrene and substituted styrenes eg. alpha-methyl styrene, alpha-olefins, eg. C₁₂-alpha-olefin, vinyl ethers, eg. butyl vinyl ether; allyl ethers, eg. butyl allyl ether; unsaturated hydrocarbons, eg ethylene, propylene, cyclohexene and dienes, eg. 1,6 hexadiene, divinyl benzene, cyclohexadiene.
4. A composition according to claim 1, 2 or 3, wherein the polymeric thickener comprises a copolymer of methacrylic acid and styrene, preferably having proportions (wt %) of methacrylic acid and styrene ranging from 80:20 to 20:80.
5. A composition according to any one of the preceding claims, wherein the polymeric thickener is substantially non cross-linked.
6. A composition according to any one of the preceding claims, wherein the polymeric thickener has a molecular weight of about 100,000.
7. A composition according to claim 6, wherein the polymeric thickener comprises 54 wt% methacrylic acid and 46% styrene, with a molecular weight of about 100,000 and substantially no cross-linking.
8. A composition according to any one of the preceding claims, wherein the polymeric thickener is present in an amount from 0.1 to 7.5% by wt as active material, preferably 0.5 to 3% by wt as active material.
9. A composition according to any one of the preceding claims, wherein the bleach material comprises hypochlorite or compounds producing hypochlorite in aqueous solution.
10. A composition according to claim 9, wherein the bleach material comprises sodium hypochlorite, preferably in an amount ranging from 0.2 to 15% by weight, more preferably 0.2 to 10% by weight.
11. A composition according to any one of the preceding claims, wherein the surfactant comprises a mixture of primary alkyl sulphates (PAS) and linear ether sulphates (LES), conveniently in wt proportions of active matter of PAS:LES of 2:1.
12. A composition according to any one of the preceding claims, wherein surfactant is present in an amount in the range 0.5 to 5% by wt active matter, preferably about 2% to 3% by wt active matter.
13. A composition, according to any one of the preceding claims, further comprising electrolyte.
14. A composition according to any one of the preceding claims, wherein the bleach comprises hypochlorite and the polymeric thickener comprises 54% methacrylic acid and 46% styrene with a molecular weight of about 100,000, being substantially non-cross-linked, and the surfactant further comprises one or more lauryl ether sulphates.

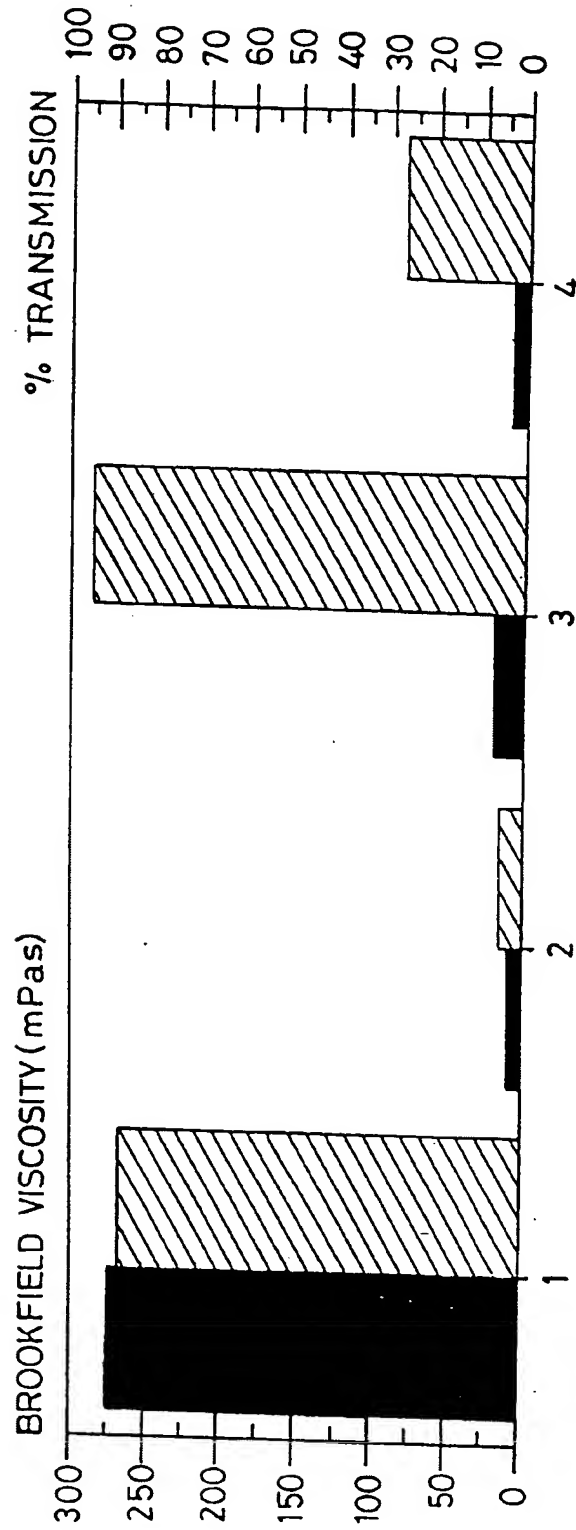


Fig. 1

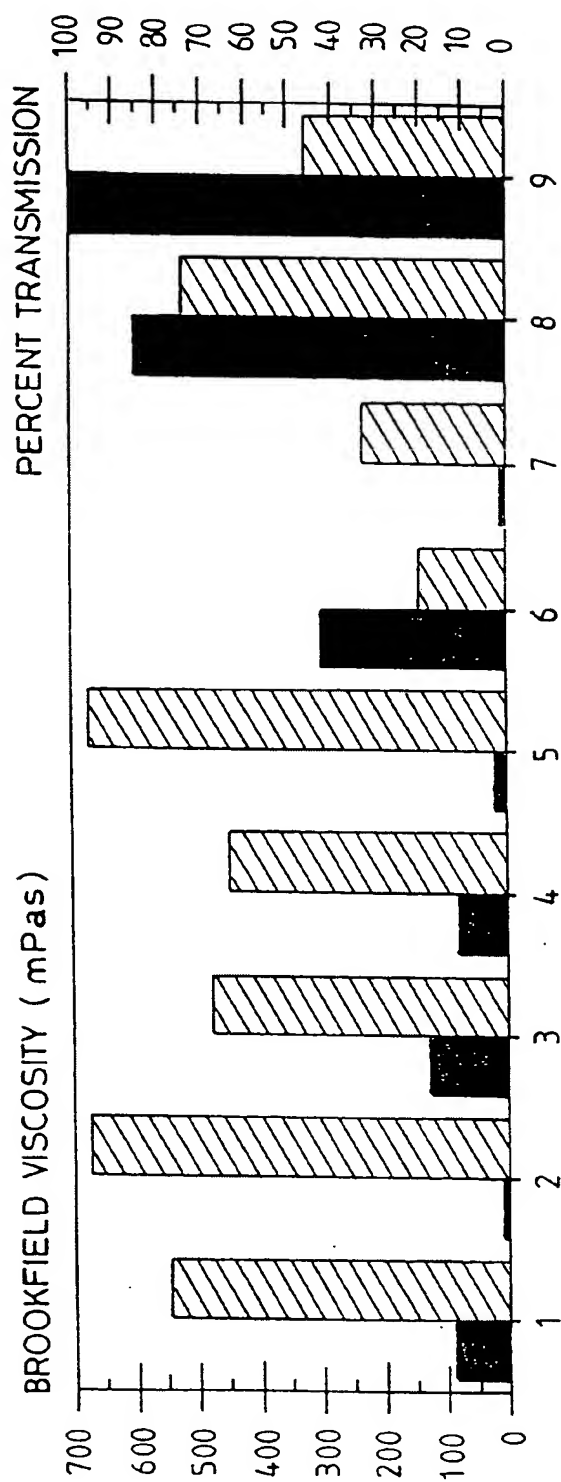


Fig. 2



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(11) **EP 0 636 689 A3**

(12) **EUROPEAN PATENT APPLICATION**

(88) Date of publication A3:
01.05.1996 Bulletin 1996/18

(51) Int Cl.⁶: **C11D 3/39, C11D 3/395,
C11D 3/37, C11D 1/14**

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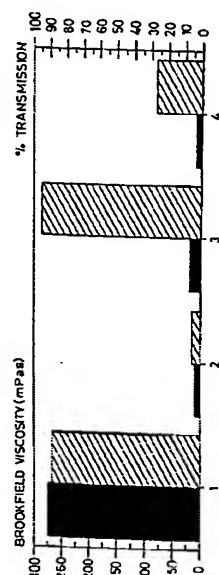


Fig. 1

EP 0 636 689 A3



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EUROPEAN SEARCH REPORT

Application Number
EP 94 30 5450

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
Y	WO-A-91 16409 (UNILEVER PLC ;UNILEVER NV (NL)) * page 3, line 11-22 * * page 11-12 * * page 13, line 3-5 * * page 13, paragraph 13-22 * * claims *	1-14	C11D3/39 C11D3/395 C11D3/37 C11D1/14
D,Y	EP-A-0 256 638 (CLOROX CO) *abstract* * page 8, line 17-30 * * page 9, line 32-35 * * page 12, line 20-35 * * page 13, line 27 * * claims 1-5 *	1-14	
A	GB-A-1 360 909 (JOHNSON & SON INC S C) * the whole document *	1-14	
A	WO-A-91 12307 (UNILEVER PLC ;UNILEVER NV (NL)) * page 2, line 1-29 * * page 5, line 17 * * page 10, line 29-35 * * page 11, line 5-24 *	1-8,12,13	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C11D
P,A	EP-A-0 574 086 (COLGATE PALMOLIVE CO) * the whole document *	1	
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 22 February 1996	Examiner Pelli Wablat, B
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

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